Germanium Directional Solidification in United States Microgravity Laboratory-2

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Special Notice: *This research task is being performed as a part of the cooperative agreement for microgravity research between MSFC, the Universities Space Research Association, and the University of Alabama, Huntsville. The principle investigator for the investigation is:*
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The primary purpose of this task is an assessment of the change from crystal growth furnace (CFG) to gravity gradient (GG) attitude during the second United States Microgravity Laboratory (USML–2) mission in an otherwise steady-state growth system using the demonstrated capabilities of the interface demarcation technique which are briefly outlined below. Thus, it will be possible for the first time to quantify a tolerable deviation of the space shuttle microgravity environment from ideal microgravity conditions. This information should prove useful in defining parameters for future materials processing in space.

Materials processing in space is an important aspect of the commercial utilization of the space environment by taking advantage of the tremendous reduction of the gravitational force acting on a crystal growth system (microgravity environment), representing a decisive advantage for producing high-quality crystalline materials. The production of such high-quality crystals in space processing invariably implies defect-free large crystals without compositional inhomogeneities.

The growth and solidification of materials implies a temperature gradient across a solid/melt interface in pure materials, and additional solute concentration gradients in doped semiconductors. However, such temperature and solute gradients in a normal gravitational field induce free convection in the melt, and the accompanying generation of changes in density and latent heat of fusion at the interface strengthen or weaken these gradients.

These gradients and the propagation velocity of the interface are coupled in a complex way to the shape of the solid/melt interface during the solidification process. This dictates strict control of the thermal and solute environment in the melt, crystal, and the containment ampoule inserted in the processing cartridge in the CGF. Hence, it is mandatory to develop a thorough understanding of the thermal properties involved in the solidification process prior to attempting any correlation of a ground-based crystal growth experiment to its duplicate in a microgravity environment.

Gallium-doped single-crystalline germanium was chosen as a model substance since its thermo-physical properties are well understood. In this growth system, the passage of well-defined electrical pulses of approximately $20A/cm^2$ current density across the solid-melt interface of the solidifying crystal introduces localized changes in the dopant segregation behavior at the interface (interface demarcation).

Although nontrivial — the heights of the interface demarcation lines to be observed are in the nanometer range — differential interference contrast optical techniques fairly readily resolve these features on highly polished and carefully etched crystal segments grown, and thus allow the determination of the shape of the interface. With knowledge of the pulse repetition rate, these interface demarcation lines can be used to measure the instantaneous microscopic growth rates throughout the grown material. The accompanying microphotograph (fig. 120) demonstrates the power of this technique: It shows a segment of the

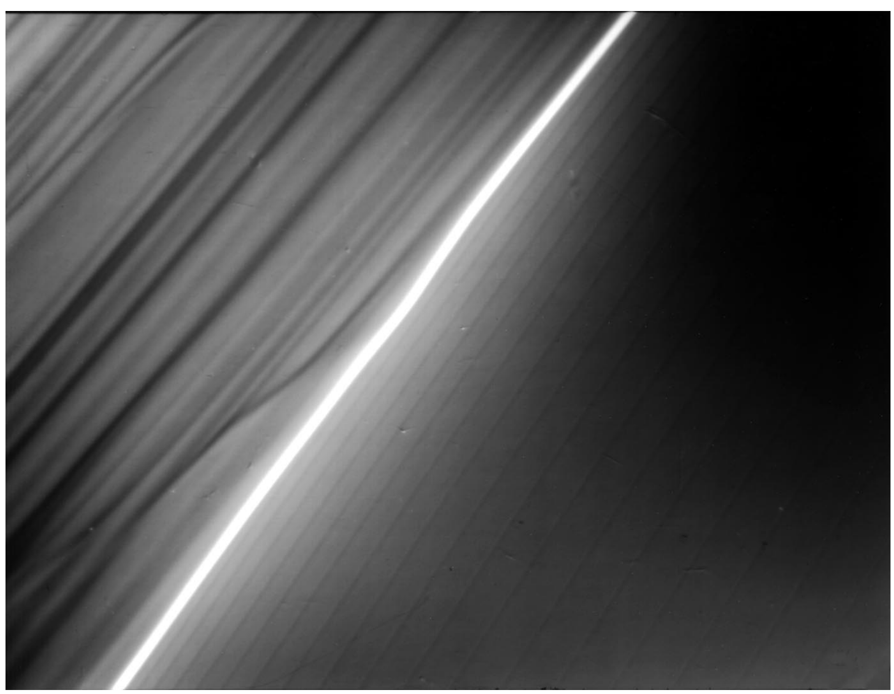

FIGURE 120.—Meltback interface.

initial 15 min of regrowth with a portion of the Czochralski grown rotational striations, the original meltback interface as a narrow white band, and the increasingly spaced interface demarcation lines.

Complemented by high-resolution electrical measurements (spreading resistance profiling) which yield information on localized dopant concentration changes, instantaneous growth rate changes can be correlated with dopant segregation behavior. This information can be used in the solution of the equations describing solidification processes in terms of heat transfer and fluid dynamics: First, the so-called Stefan problem in which solely the effects of the thermal gradients on the speed and shape of the crystal/melt interface are analyzed. Next, the combined effects of both the solute gradients and the temperature gradients are analyzed. In addition, the formation of morphological instabilities due to small departures from the theoretical limits of the energy and mass fluxes must also be examined, since they can lead to the formation of non-planar or even unstable oscillatory interfaces. Finally, the “rubber band effect”, i.e. the degree of coupling of the growth system with the furnace, both at the onset and during directional solidification using these techniques must be analyzed, and explained.

Preliminary results on one of the crystals grown during mission USML–2 which included a translation arrest during a period of 90 min, i.e. one orbital period, clearly define the change over from steady-state growth conditions to stationary fluctuations, followed by a transient growth region upon resumption of growth. These results also indicate a very gradual change in the interface shape upon completion of the change of attitude maneuver. A three-dimensional reconstruction of this event is in progress.

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Biographical Sketch: Charles Baugher is a materials scientist and deputy division chief in the Microgravity Science and Applications Division of Space Sciences Laboratory. His recent research has been in the area of defining the low-level acceleration environment of the Space Shuttle during microgravity experimentation and in studying effects of that environment on materials processing. He has been published in the areas of electromagnetic propagation in plasmas, the interactions of plasmas with spacecraft, astronomical observations in the infrared, and the morphology of the Earth’s magnetosphere.